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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:  
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.  
If no title is shown please refer to the description.  
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Strengthened ceramic restoration

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Title: Strengthened ceramic restoration

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The present invention relates to an aesthetic ceramic dental restoration, and particularly to a crown, part-crown or a bridge. In addition, the invention relates to a process for manufacturing such a product. More specifically, the present invention concerns a process for the production of an  
5 aesthetic heat-pressed restoration in occlusion, which is strengthened by a zirconia suprastructure, as well as the product obtainable in this way.

Dental restorations are mostly metal-ceramic composite structures, comprising a metallic framework used for load bearing, and ceramic or porcelain coatings for aesthetic appearance. More in detail, nowadays, about  
10 80% of all fixed restorations are metal-ceramic restorations; these metal ceramic restorations show a clinical survival rate of at least 95% after 7.5 years.

In the vast majority of manufacturing processes, the ceramic material is applied by using repeated layer formation. Particularly, a layer of  
15 ceramic or porcelain is applied, followed by firing or sintering, which steps are repeated until the suitable dental restoration is obtained. During this conventional process, each consecutive layer of porcelain shrinks during sintering. This shrinkage makes it very hard to obtain proper occlusal contacts with the antagonist teeth.

20 It would be very desirable to find a cost and time saving process compared to the traditional porcelain layering process.

Another disadvantage of the conventional layering technique is that often defects such as bubbles or clefts are formed, having an adverse effect on the adhesion between porcelain and either translucent layer or core or support  
25 material.

In the prior art, attempts have been made to solve or reduce these problems. In this light, reference can be made to an article titled "Gießen,

pressen, modellieren" published in DZW (Woche) 23/02. In this reference, a process is described for preparing a dental restoration in occlusion, wherein a cast metal structure is first covered with two opaque liner layers, followed by waxing-up up till the occlusal contacts with the opposing teeth and mesio-  
 5 distal contacts with the neighbouring teeth are obtained. Sprues are attached to the wax part, the restoration is embedded in a refractory material and a tooth coloured glass ceramic is pressed onto the metal prestructure. Because of the non-translucent nature of the metal, the metal shimmers through the translucent outer glass ceramic which results in an less attractive looking  
 10 restoration.

Beside the fact that these known metal-ceramic combinations show limitations concerning aesthetic appearance, the last years, the use of metals in the oral cavity has been disputed due to their biological incompatibility risk. This led to the need for bio-inert, metal free dental restorations.

15 In the art, it was proposed to meet this need by making all-ceramic dental restorations or prostheses. Such all-ceramic dental restorations have been made of feldspathic porcelain, leucite re-inforced porcelain, alumina, glass-infiltrated porous alumina and glass ceramics. These ceramic materials show, however, low bend strengths and toughness, which properties imply  
 20 design restrictions, non-reliability and complicated multistep manufacturing procedures for, *e.g.*, dental bridges.

Based on these findings, the person skilled in the art has focussed its attention to zirconia base materials to replace the metal base, because zirconia has promising properties in respect of durability and longevity.  
 25 Reference can be made to the article of Filser *et al.* titled "All-Ceramic Dental Bridges by Direct Ceramic Machining (DCM)" as published in: Materials in Medicine, Materials Day, Department of Materials, Eds. M.O. Spedel; P.J. Uggowitzer; vdf Hochschulverlag AG, ETH Zürich; Zürich (1998) 165-189  
 Zirconia structures can be produced by slib casting or by milling. Because of

the opaqueness of zirconia, a tooth-coloured dental glass is used to bring the proper natural aesthetics to the zirconia base.

The zirconia used is mostly partially stabilized zirconia, and more in detail tetragonal zirconia stabilized with yttria, which has high strength and  
5 toughness.

The starting powder for these ceramic restorations has special demands for particle size and morphology as described in detail in the article of Filser *et al.*. These demands are necessary for an homogeneous result on the consolidation by isostatic pressing at a pressure higher than 2000 bar. Blocks  
10 are subsequently partially sintered at 900°C until the powder particles are bonded by neck growth to give the ceramic body strength high enough to be able to mill it. Milling is done taking account of an enlargement factor that corresponds to the expected sintering shrinkage in the final sintering step. The zirconia structure is then coated with a veneer porcelain to meet the patient's  
15 requirements concerning colour and translucency.

Although the traditional layering technique could be used, thermal pressing of dental glass into a lost wax form is a more effective and economic way.

Cornelissen gives in TTM: Magazine voor Tandartsen en  
20 Tandtechnici 10 (2001) and in Quintessenz Zahntech. 28(2) (2002), 150-158, a description of the Cordent crown. This crown is prepared by directly modelling the entire dentine form inclusive crown shoulder to an AGC Galvano yellow cap, embedding in refractory, pressing ceramic, and debedding, followed by divesting and glazing firings. The yellow metal cap is said to provide a nice  
25 deep orange glow at the occlusal and near the edges. Cornelissen notes that in the Cordent crown the advantages of all-ceramic systems are combined with the advantages of metal-ceramic systems.

Processes are described whereby a wax model is embedded in a refractory material and after hardening of the mould the wax is burnt-out. A  
30 glass material in the form of a dense pellet is brought in over the pressing

connector channels and with a refractory cylinder under thermal plastification the pellet is pressed in the mould.

These materials lack sufficient strength to be used for more stressed applications such as bridges.

5 In accordance with the present invention full-ceramic dental restorations are prepared which meet all needs sketched combined with the advantages of the described prior art systems. Other advantages and benefits of the present invention will become clear after reading the following description.

10 More in detail, the present invention relates to a process for the preparation of a full-ceramic dental restoration, which should be in occlusal contact with opposing teeth and in mesio-distal contact with neighbouring teeth, comprising heat pressing of a tooth coloured pressing glass on a fully or partially supporting structure of yttria doped tetragonal zirconia (YTZP),  
15 comprised of yttria doped tetragonal zirconia ceramic having a grain size, as measured by the linear intercept method, of less than  $0.6\ \mu\text{m}$ , wherein the thermal expansion coefficient (TEC) of the pressing glass lies between 9.0 and  $11.0\ \mu\text{m/m.K}$  (measured in the range of from 25 to  $500^\circ\text{C}$ ) and the pressing temperature of the pressing glass lies between 750 and  $1000^\circ\text{C}$ .

20 In a further aspect, the present invention relates to a full-ceramic dental restoration, comprising a fully or partially supporting structure of yttria doped tetragonal zirconia (YTZP) and a heat pressed tooth coloured pressing glass, which restoration is modelled to be in occlusal contact with opposing teeth and in mesio-distal contact with neighbouring teeth. Preferably, this full-  
25 ceramic dental restoration is obtainable by the process of the present invention.

Surprisingly, it was found according to the present invention that a tooth coloured glass ceramic when pressed on a strong zirconia structure, which has a certain degree of translucency, a very naturally looking  
30 restoration can be obtained, even when no liner was used. The zirconia used is

referred to in the above-mentioned article of Filser *et al.*, and described in more detail by Lüthy in W.H. Mörmann (ed.), CAD/CIM in Aesthetic Dentistry, Quintessenz, Chicago, (1996), 229 ff., and has a high strength and can be used for support structures in single element restorations as well as in larger constructions such as 3 to 4-unit bridges.

The ability to heat-press a tooth coloured glass ceramic onto such a structure means an enormous time saving when compared to the layering of porcelain powder and subsequently sintering the powder as is usual in the traditional way. This cost and time saving is in comparison to the tradition  
10 porcelain layering process. During this latter process each consecutive layer of porcelain shrinks during sintering making it very hard to obtain proper occlusal contacts with the antagonist teeth.

Because the press-pellet is already in a tooth colour, the colour of the restoration will not vary from the given colour as can be the case when  
15 layering with a variety of colours. The colouring of the porcelain used in the present invention is known to the person skilled in the art. A suitable method is described in detail in DE-OS-199 04 522, which document is incorporated by reference in the present description for describing the method of colouring.

It is for instance possible to apply pure oxidic pigments on the  
20 zirconia, followed by the sintering together of the zirconia and oxidic pigments. In a particular embodiment, the pigments are pressed with a binder into a block or cylinder to be used as a pensil to bring the pigments on the zirconia surface.

In another embodiment, the partially sintered zirconia structure is  
25 impregnated with a solution of metal chlorides, nitrates, acetates or alcoholates and, subsequently dried and sintered to obtain a tooth coloured zirconia structure after sintering. Very suitable results are obtained, while using metals of the group of iron, praesodimium, nickel, cerium, erbium, cobalt, and copper.

In a preferred embodiment, the supporting structure is densely sintered; preferentially, the ceramic is produced by CAD/CAM technology. More specifically, in a preferred embodiment of the process of the invention the ceramic is milled by a CAD/CAM-system in the green state or in a partially  
5 sintered state, followed by sintering to full density.

In the process of the invention very good results are obtained with a supporting structure formed from electrophoretic deposition of zirconia from a slurry, followed by sintering to full density.

In another embodiment, the structure is milled out of an hot  
10 isostatically hopped zirconia.

Apart from the above, the German "Patentschrift" 196 30 412 teaches a process for the fabrication of a full-ceramic dental build-up on a zirconia root pin, wherein a zirconia-glass is heat-pressed against the root pin that has a TEC that is the same or up to  $3.0 \mu\text{m}/\text{m.K}$  higher than the TEC of  
15 the zirconia glass. This prior art document also describes the build-up with zirconia glass of a tooth replacement without mentioning how the zirconia is used. No reference is made to occlusal restorations, as is the subject of the present invention. Moreover, the zirconia glass described in the German patent has low transparency and cannot be used for aesthetic tooth-like  
20 restorations, as is the case with the present invention.

The process of the invention directly presses the occlusal contacts in an aesthetically working material after the occlusal contact with the antagonistic teeth and the mesio-distal contacts with the neighbouring elements having been precisely modelled in wax in the traditional way.

25 Another advantage over the traditional layering is the high density, low defect structure that can be obtained by pressing a densely sintered glass instead of applying a porcelain as a powdery substance with subsequent sintering. The latter shows very seldom a bubble-free structure.

Another advantage over the traditional layering is the adhesion  
30 obtained between the translucent material and the zirconia core. During



layering the boundary layer often shows defects such as bubbles and clefts, while the pressed on glass shows an excellent defect free boundary, resulting in a better adhesion and a higher structural strength.

For particular embodiments, advantages can be obtained when a  
5 conventional liner is applied to the supporting zirconia structure which liner has a melting point less than 50°C lower than the pressing temperature of the pressing glass.

The process offers another advantage in the possibility of directly  
pressing a shoulder with a perfect fitting margin, without showing the  
10 substructure at the edge of the supporting material. For this the edge of the supporting core is kept short by 0.5 to 2 mm from the edge.

The process of the invention has the advantage of the possibility to  
create a chameleon effect, when using the heat press ceramic as shoulder  
material (Figure 3; Right). Because the wax has been applied after the zirconia  
15 structure is fitted on the gypsum die, the wax-up for the shoulder can follow the die exactly around the margin, and the subsequently pressed shoulder reproduced in glass ceramic will have the same good fit. In the traditional way involving several steps of layering porcelain, several corrections with porcelain additions have to be made to produce a fitting margin, because of the  
20 shrinkage of the porcelain powder during sintering.

In a particular aspect of the process of the invention, the structure  
is, hence, kept short by 0.5 to 2.0 mm from the edge of the final restoration to  
be made, after which the pressing glass is pressed in such a way that it forms  
an aesthetically pleasing shoulder without showing the core at the margin.

25 Preferably, the stresses are transferred on the core only, while leaving the shoulder free from the prepared tooth.

The present inventors have found that the stability of the adhesive  
bond strength between the glass and the zirconia is critically dependent upon  
the susceptibility of the zirconia material to low temperature degradation  
30 (LTD). Although YTZP zirconia ceramics are known to have a high strength

and toughness, they are also known to be susceptible to strength degradation upon exposure to steam in the temperature range of about 100-500°C. The origin of this LTD phenomenon is attributed to a reaction involving water and the Zr-O-Zr bonds of the ceramic. This reaction causes transformation of zirconia grains from their desired tetragonal state to the monoclinic state. This transformation is accompanied by a volume expansion in the transformed grain of about 4 vol.%, which causes microcracking in the component and, accompanied, strength degradation.

Without wishing to be bound by any theory, it is believed that the environmental conditions present in the mouth are such that LTD may occur in the zirconia dental components and that this phenomenon may have a negative impact on the strength of the YTZP-dental glass bond. In particular, the temperatures in the mouth are typically simulated by thermal cycling between about 5 and 55°C. Although these temperatures are somewhat below those typically associated with the LTD phenomenon, Chevalier *et al.* (see: Bioceramics 10 Ed. L. Sedel and C. Rey (Proc. of the 10<sup>th</sup> Int. Symp. on Ceramics in Medicine, Paris, France, October 1997) Elsevier Science Ltd.) have suggested that LTD may also occur in some YTZP zirconias at temperatures as low as about 37°C. Thus, it is believed that LTD may act upon YTZP in dental systems. With regard to YTZP-glass bonds, it is believed that LTD of the YTZP may cause general microcracking in the vicinity of the transformed grain and in particular at the uncracked surface of YTZP material, and that this microcracking degrades the adhesive bond strength of the glass-zirconia system and allows for further ingress of water into the zirconia material, thereby accelerating the spread of LTD.

The selections made in the process of the present invention prevent or at least inhibit or reduce LTD.

In said article of Chevalier *et al.* suitable yttria-stabilised zirconias for use in the present invention are identified.

Very good results are obtained in a process, wherein the connector to the press pellet reservoir and the specimen is formed by a continuous flow plate of 1.5 to 2.5 mm thick. This embodiment will be described in more detail in Figure 8 and the accompanying text.

5           The present invention will now be described in more detail, wherein reference is made to the drawings, wherein:

Figure 1 shows a schematic overview of a zirconia structure with wax-up;

Figure 2 shows the zirconia structure of Fig 1 after pressing;

10          Figure 3 shows final restorations with a liner and with a zirconia shoulder (prior art) and with a shoulder of heat-pressed ceramics in accordance with the present invention;

Figure 4 shows a flow scheme for the production of zirconia structures to be used in the present invention;

15          Figure 5 shows a flow scheme for the production of the pressing step wherein pressing glass is brought on the zirconia structures according to the present invention;

Figure 6 shows a microphotograph of a dental glass pressed to YTZP zirconia in the absence of a liner;

20          Figure 7 shows a microphotograph of a dental glass pressed to YTZP zirconia covered with a liner; and

Figure 8 shows a special flow plate for a pressed glass structure.

25          All of the above mentioned advantages are provided by the present invention which comprises the process of producing an esthetic heat-pressed restoration in occlusal and mesio-distal contact with neighbouring and opposing teeth, strengthened by a fine grained yttria doped tetragonal zirconia (YTZP) support structure.

30          The new glass composition that was produced has a pressing temperature from about 750 to 1000°C, and preferably from 900 to 950°C and

has a coefficient of thermal expansion of from about 9.0 to 11.0, and preferably from about 9.0 to 10.0  $\times 10^{-6}/^{\circ}\text{C}$  ( $25^{\circ}\text{C}$  to  $500^{\circ}\text{C}$ ).

More specifically, the present inventors have found a low expansion glass or preferably a glass-ceramic material suitable for over-pressing a zirconia suprastructure such as a crown, part-crown or bridge has been developed.

Taking into account the guidances given herein above, the glass used in the process of the invention preferably has the following chemical composition: 7-15 wt.%  $\text{Al}_2\text{O}_3$ , 13-23 wt.% of ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ), 1-3 wt.% of ( $\text{BaO} + \text{CaO}$ ), 1-3 wt.% ( $\text{Sb}_2\text{O}_3 + \text{Li}_2\text{O}$ ), and 0.2-1.2 wt.% fluor, the balance being  $\text{SiO}_2$ , and colouring compositions. In a more preferred embodiment, the glass has the following chemical composition: 7-15 wt.%  $\text{Al}_2\text{O}_3$ , 6-14 wt.%  $\text{K}_2\text{O}$ , 5-11 wt.%  $\text{Na}_2\text{O}$ , 0.2-2.5 wt.%  $\text{BaO}$ , 0.1-1.5 wt.%  $\text{CaO}$ , 1.2-2.5 wt.%  $\text{Sb}_2\text{O}_3$ , 0.05-0.5 wt.%  $\text{Li}_2\text{O}$ , and 0.5-1.0 wt.% fluor, the balance being  $\text{SiO}_2$ , and colouring compositions.

Low expansion glasses for use in the present invention can be produced by blending powdered metal oxides or carbonates or nitrates in the appropriate proportions. The blended powders are fused to form a glass melt followed by quenching, drying, ballmilling and sieving by means known in the art.

The powder formed from these glasses have a particle size of preferably less than 106  $\mu\text{m}$ ; they are pigmented to obtain a toothlike appearance. Then the powder is granulated with a binder and uniaxially dry-pressed at room temperature and then sintered at a temperature of  $800^{\circ}$  to  $1000^{\circ}\text{C}$ ., preferably  $900^{\circ}$  to  $960^{\circ}\text{C}$ ., for 1 minute to 1 hour, preferably 1 minute to 30 minutes.

The glass-ceramic pellet obtained in this way can then be over-pressed on a zirconia suprastructure embedded in the mould to obtain a restoration in occlusal contact with the opposing teeth and mesio-distal contacts with neighbouring teeth. A suitable embedding material is silica-

based refractory such as Carrara® Press Speed (*ex* Elephant Dental B.V., Netherlands).

The zirconia supporting structure is prepared from yttria doped tetragonal zirconia. This stabilized zirconia should have a grain size, as  
5 measured by the intercept method, of less than  $0.6\ \mu\text{m}$ . Very good results are obtained when using a partially stabilized zirconia with a density of more than 99.0 wt.%, and preferably more than 99.5%, such as 99.8% of the theoretical density with an open porosity of less than 0.4% and preferably less than 0.2%. The elasticity modulus was not higher than 220 GPa and the fracture  
10 toughness was at least  $5\ \text{MPa}\cdot\text{m}^{1/2}$ .

Not suitable were partially stabilized zirconias (PSZs) such as magnesia stabilized zirconias (Mg-PSZ) or calcia-stabilized zirconias (Ca-PSZ). These zirconias are characterized by a coarsely grained structure (of the order of about  $50\ \mu\text{m}$ ) containing significant residual intragranular porosity. In  
15 addition, due to the high sintering temperature needed to sinter PSZs, impurity diffusion at the grain boundaries often produces a significant glassy phase, thereby affecting mechanical properties. The weak intergranular bonds of the coarse PSZ grains are more easily broken during adhesion testing, thereby lowering overall bond strength. In contrast, the very fine  
20 microstructure of biomedical grade YTZP materials results in very strong intragranular bonding. For this reason YTZP appeared a material providing a surface suitable for bonding to heat-pressed glass. Preferably, the YTZP ceramic has a grain size of less than  $0.5\ \mu\text{m}$ .

If desired, the zirconia structure can be coloured by ionic or complex  
25 containing solutions which contain rare earth elements or elements of the adjacent group. The partially sintered zirconia structure can for instance be dipped in such a solution, dried and sintered to its final density.

Figure 4 shows a production flow scheme for the zirconia structures for use in the present invention. Particularly, the zirconia powder is subjected  
30 to isostatic pressing, preferably using CAD/CAM technology. The pressed form

is subjected to partial sintering and subsequently milled, although it can also be milled in green state, preferably using a CAD/CAM system. After milling the oversize, the formed structure is sintered to full density giving the zirconia substructure.

5           This substructure is further treated following the scheme shown in figure 5. The substructure and a wax-up occlusion (see Figure 1) are invested in refractory. The wax is burnt out, and the mould is preheated. Glass pellets are pressed in the mould, after which the investment is removed (see Figure 2). The product formed can be subjected to a treatment to glaze its surface.

10           In Figure 3, two final restorations of the present invention are described. The left-hand image shows a zirconia shoulder structure coated with a liner on which a low expansion glass of the invention is pressed. Thereto a suitable liner, for instance one consisting of 54.8 wt.%  $\text{SiO}_2$ , 12.9 wt.%  $\text{Al}_2\text{O}_3$ , 11.5 wt.%  $\text{K}_2\text{O}$ , 8.7 wt.%  $\text{Na}_2\text{O}$ , 10.4 wt.%  $\text{CeO}_2$ , 1.0 wt.%  $\text{Li}_2\text{O}$  and  
15           0.4 wt.%  $\text{B}_2\text{O}_3$ ; or one consisting of 58.5 wt.%  $\text{SiO}_2$ , 12.6 wt.%  $\text{Al}_2\text{O}_3$ , 11.0 wt.%  $\text{K}_2\text{O}$ , 7.1 wt.%  $\text{Na}_2\text{O}$ , 10.4 wt.%  $\text{CeO}_2$ , 0.4 wt.%  $\text{Li}_2\text{O}$ , was applied in a single coat of 20 to 40  $\mu\text{m}$  onto a densely sintered zirconia support-structure and fired at 800°C and 915°C for both liners illustrated respectively.

20           The liner-coated zirconia coping was waxed up and sprued as described before. The substructure comprising the liner was subsequently overpressed with the glass material.

          The right-hand image of Figure 3 shows a zirconia substructure with a shoulder in heat-pressed glass ceramics.

          It was additionally found that when a special flow plate (fig 8) was  
25           used to guide the flowing heat-pressed glass a better filling was obtained with less material, that with separate sprues as is usual in the traditional technique. The plate is 1.5 to 2.5 mm thick and starts from one side with the press pellet reservoir and is over its full length in contact with the pressed specimen. The flow plate is easier to separate with the appropriate cutting  
30           wheels than the much thicker separate sprues in the traditional technique.

The plate makes contact with the pressed specimen in a place where it does not interfere with the contact surfaces to the antagonistic teeth and the mesio-distal contact areas.

5 The present invention will be illustrated in more detail in the following, non-limiting examples. Where reference is made to percentages, weight percentages drawn to the weight of the final composition are meant, unless otherwise indicated.

#### Examples 1-4

10

Low expansion glasses or glass-ceramic materials suitable for over-pressing a zirconia suprastructure such as a crown, part-crown or bridge were prepared. Thereto, four mixtures were produced by blending powdered metal oxides or carbonates or nitrates in the appropriate proportions. The blended  
15 powders were fused to form a glass melt followed by quenching, drying, ballmilling and sieving by means known in the art (see Table 1 for the final compositions).

The powder formed from either one of the four undermentioned glasses having a particle size of less than 106  $\mu\text{m}$  are pigmented to obtain a  
20 toothlike appearance. Then the powder was granulated with a binder and uniaxially dry-pressed at 900°C for 20 minutes.

A partially stabilized zirconia with a density of 99.8% of theoretical density with an open porosity of less than 0.2% was obtained by following the method depicted in figure 4. The elasticity modulus was about 200 GPa and  
25 the fracture toughness was about 5  $\text{MPa}\cdot\text{m}^{1/2}$ .

The glass-ceramic pellets obtained as described were over-pressed on the zirconia suprastructure embedded in a mould to obtain a restoration in occlusal contact with the opposing teeth and mesio-distal contacts with neighbouring teeth.

30

Table 1. Composition of heat-press glass ceramic

Component	Ex. 1	Ex. 2	Ex. 3	Ex. 4
SiO <sub>2</sub>	66.2	65.0	67.4	66.0
Al <sub>2</sub> O <sub>3</sub>	10.7	7.5	13.9	14.4
K <sub>2</sub> O	9.5	12.2	6.8	9.1
Na <sub>2</sub> O	8.5	9.6	7.4	6.8
BaO	1.2	2.0	0.4	0.4
CaO	0.7	0.2	1.3	1.1
Sb <sub>2</sub> O <sub>3</sub>	2	2.3	1.8	1.5
Li <sub>2</sub> O	0.2	0.4	0.1	0.2
F	0.8	0.9	0.7	0.6
Pressing temperature, °C	900	800	940	930
Thermal Coefficient of Expansion (TCE), $\times 10^{-6}/^{\circ}\text{C}$ (25°C to 500°C)	9.5	11.0*	8.7	10.1
Break strength of incisor tooth, N (without liner)	5200	5800	6300	6200
Break strength of incisor tooth, N (with liner B)	N.D.	N.D.	N.D.	6000
Thermal Shock Tests, nr. of cycles up to 20 until cracks appear	20	Cracks	Cracks	20

\* TCE was measured from 25°C to 400°C

N.D. = not determined

5

A liner material A, consisting of 54.8 % SiO<sub>2</sub>, 12.9 % Al<sub>2</sub>O<sub>3</sub>, 11.5 % K<sub>2</sub>O, 8.7 % Na<sub>2</sub>O, 10.4 % CeO<sub>2</sub>, 1.0 % Li<sub>2</sub>O and 0.4 % B<sub>2</sub>O, was applied in a single coat of 20 to 40  $\mu\text{m}$  onto a densely sintered zirconia support-structure and fired at 800°C.

10

The liner-coated zirconia coping was waxed up and sprued as described before.

Upon overpressing a zirconia-structure embedded in a silica-based refractory form (Carrara® Press Speed, Elephant Dental B.V., Netherlands) with densely sintered pellets having the oxidic composition of Ex. 4, the liner layer melted and dripped to the margin area.

15

Another liner material B, consisting of 58.5 % SiO<sub>2</sub>, 12.6 % Al<sub>2</sub>O<sub>3</sub>, 11.0 % K<sub>2</sub>O, 7.1 % Na<sub>2</sub>O, 10.4 % CeO<sub>2</sub>, 0.4% LiO<sub>2</sub> was applied in a single coat of 20 to 40  $\mu\text{m}$  and fired at 915°C. After the liner was overpressed using the



same pellets as described above testing the liner material A, the layer remained in place and good results were obtained regarding both the thickness of the liner over the whole surface of the zirconia coping as for a good esthetic appearance.

- 5           The interfaces formed were studied in cross section. The results are shown by the microphotographs in Figures 6 and 7.

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Claims

1. Process for the preparation of a full-ceramic dental restoration, comprising heat pressing of a tooth coloured pressing glass on a fully or partially supporting structure of yttria doped tetragonal zirconia (YTZP), comprised of yttria doped tetragonal zirconia ceramic having a grain size, as  
5 measured by the linear intercept method, of less than  $0.6\text{ }\mu\text{m}$ , wherein the pressing glass has a thermal expansion coefficient (TEC) of between  $9.0$  and  $11.0\text{ }\mu\text{m/m.K}$  (measured in the range of from  $25$  to  $500^{\circ}\text{C}$ ) and wherein the pressing glass has a pressing temperature of between  $750$  and  $1000^{\circ}\text{C}$ .
2. The process according to claim 1, wherein the supporting structure  
10 is comprised of densely sintered ceramic produced by CAD/CAM technology.
3. The process according to claim 2, wherein the ceramic is milled by a CAD/CAM-system in the green state or in a partially sintered state, and subsequent sintering to full density.
4. The process according to claim 1 or claim 2, wherein the supporting  
15 structure is milled out of a hot isostatically pressed zirconia.
5. The process according to claim 1, wherein the supporting structure is formed by electrophoretic deposition of zirconia from a slurry, followed by sintering to full density.
6. The process according to claim 1, wherein the structure is kept short  
20 by  $0.5$  to  $2.0\text{ mm}$  and the pressing glass is forming an aesthetically pleasing shoulder without showing the core at the margin.
7. The process according to any one of the claims 1-6, wherein a liner is applied to the supporting zirconia structure with a melting point less than  $50^{\circ}\text{C}$  lower than the pressing temperature of the pressing glass.
- 25 8. The process according to any one of claims 1-7, wherein the connector to the press pellet reservoir and the specimen is a continuous flow plate having a thickness of  $1.5$ - $2.5\text{ mm}$ .

9. Full-ceramic dental restoration, comprising a fully or partially supporting structure of yttria doped tetragonal zirconia (YTZP) and a heat pressed tooth coloured pressing glass, which restoration is modelled to be in occlusal contact with opposing teeth and in mesio-distal contact with  
5 neighbouring teeth.

10. The full-ceramic dental restoration of claim 9, obtainable by the process of any one of the claims 1-8.

Title: Strengthened ceramic restoration

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Abstract

The present invention relates to a process for the preparation of a full-ceramic dental restoration, comprising heat pressing of a tooth coloured pressing glass on a fully or partially supporting structure of yttria doped tetragonal zirconia (YTZP), comprised of yttria doped tetragonal zirconia ceramic having a grain size, as measured by the linear intercept method, of less than  $0.6 \mu\text{m}$ , wherein the pressing glass has a thermal expansion coefficient (TEC) of between  $9.0$  and  $11.0 \mu\text{m/m.K}$  (measured in the range of from  $25$  to  $500^\circ\text{C}$ ) and wherein the pressing glass has a pressing temperature of between  $750$  and  $1000^\circ\text{C}$ . In a further aspect, the invention relates to a full-ceramic dental restoration, comprising a fully or partially supporting structure of yttria doped tetragonal zirconia (YTZP) and a heat pressed tooth coloured pressing glass, which restoration is modelled to be in occlusal contact with opposing teeth and in mesio-distal contact with neighbouring teeth.

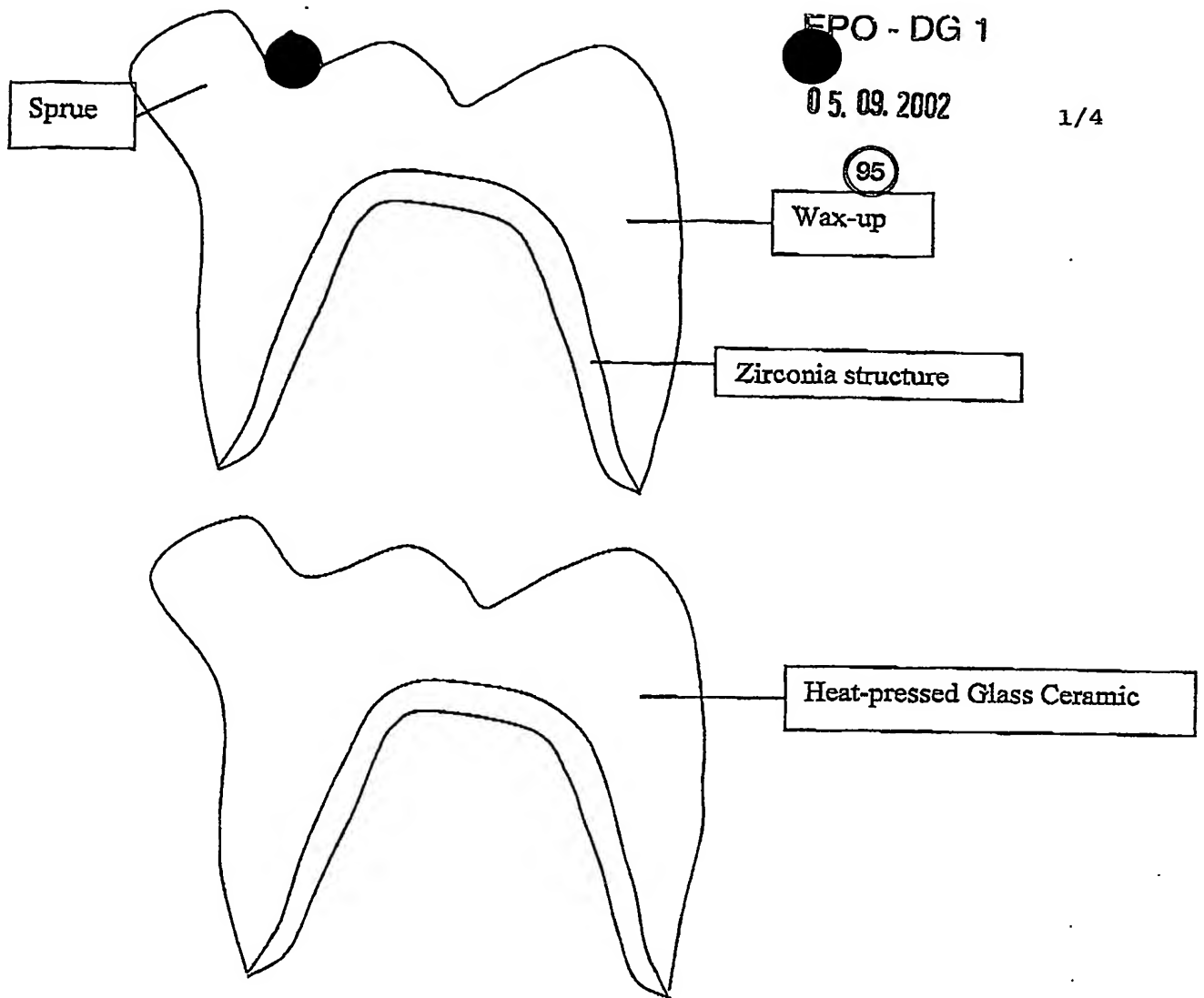


Figure 1 and 2: Zirconia structure with wax-up (1) and after pressing (2)

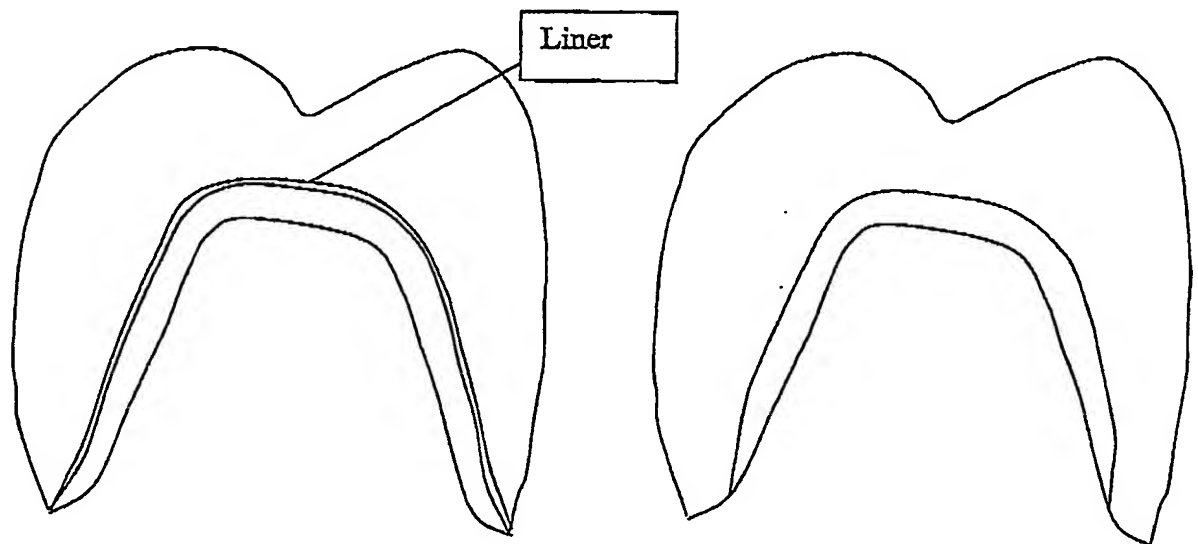


Figure 3. Left: Final restoration with a zirconia shoulder and a liner. Right: with a shoulder in heat-pressed glass ceramics.

Figure 4. Production flow for zirconia structures

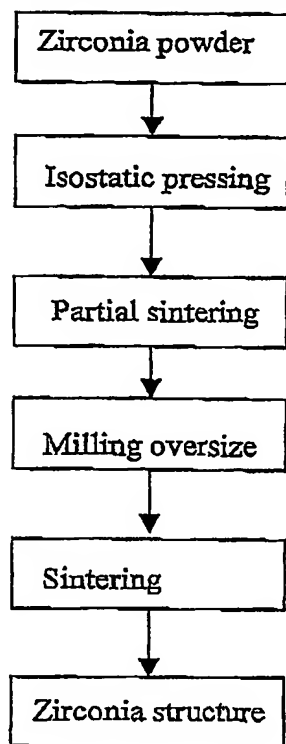
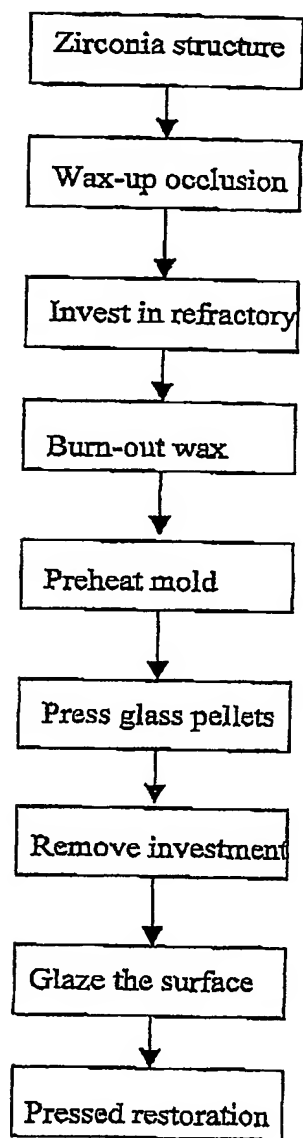


Figure 5. Production flow Press-To-Zirconia



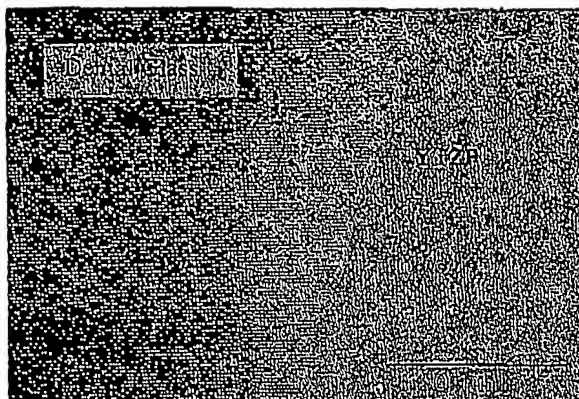


Figure 6. Dental Glass pressed to YTZP zirconia without the use of a liner

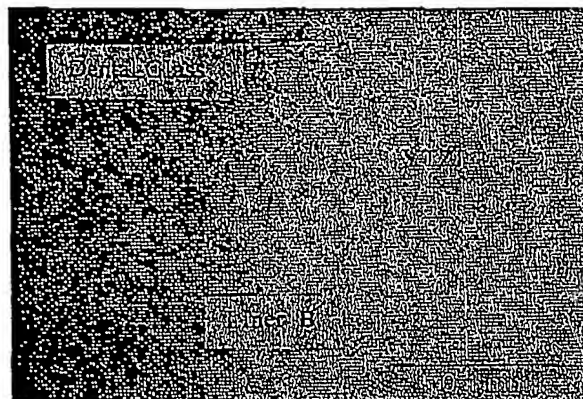


Figure 7. Dental Glass pressed to YTZP zirconia covered with liner B

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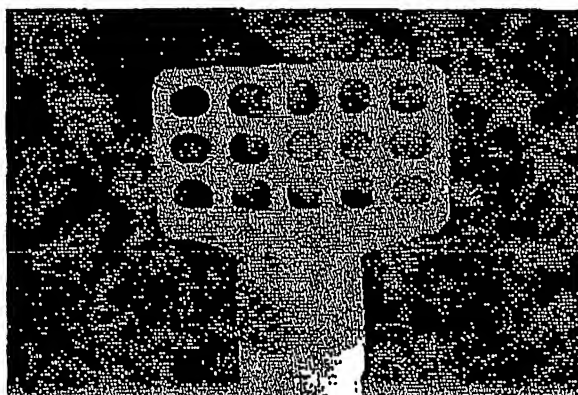


Figure 8. Special flow plate to a pressed glass structure



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